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GB 2212505 A **EP 0388481 A1** **EP 0009109 A1**
US 4971726 A
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(54) Intumescent materials

(57) An intumescent material comprises expandable graphite and is characterised by a binder incorporating a diene monomer, e.g. polynorbadene. The expandable graphite is preferably intercalated or oxygenated graphite. A method of making an intumescent material is characterised by mixing a diene monomer and discrete particles of expandable graphite. The ingredients may be mixed in a dry, or at least solid, state. Thus the diene monomer may be in granular or powder form.

A method of making an intumescent fire seal is characterised by the step of co-extruding an intumescent material as described and at least one thermoplastic material.

GB 2317616 A

TITLE: INTUMESCENT MATERIAL

DESCRIPTION

TECHNICAL FIELD

The invention relates to intumescent material comprising expandable graphite and a binder.

BACKGROUND ART

It is known from GB-B-2212505 to provide an intumescent material comprising a flexible polymeric or elastomeric binder containing expandable graphite and plasticizer. It is also known from GB-B-2212505 to provide an intumescent material comprising an intumescent material comprising expandable graphite in a flexible polymeric or elastomeric binder and made by preparing a mixture of the expandable graphite, the binder and water and causing or allowing the mixture to harden. It is further known from GB-B-2212505 to provide an intumescent material comprising

an expandable graphite in an elastomeric binder and made by preparing a mixture of the expandable graphite and a liquid emulsion or dispersion of the elastomeric binder and causing or allowing the mixture to cure by coagulation or cross-linking of the elastomeric binder.

Disadvantages of at least some known intumescent materials include moisture absorption, which adversely affects the efficiency and/or life of the material, lack of mechanical flexibility of the material when in sheet or strip form so that it tends to crack when bent, friability, and difficulties in manufacture, particularly extended drying time and lack of mechanical strength during drying, resulting in handling difficulties.

It is an object of the present invention to provide a novel intumescent material.

It is a further object of the invention to provide an intumescent material which is flexible but which does not employ a plasticizer.

It is another object of the invention to provide an intumescent material which is substantially non-hygroscopic whereby the active life of the material is extended.

It is another object of the invention to provide an intumescent material which can be made simply and quickly and which does not require an extended drying period.

DISCLOSURE OF INVENTION

According to the invention there is provided an intumescent material comprising expandable graphite and a binder incorporating a diene monomer. Preferably the diene

monomer is polynorbadene, e.g. that sold under the Trade Name "NORSOREX" by CdF Chimie S.A. of Paris, France. The expandable graphite may be of the kind sold by Industrial Adhesives Limited or that sold by Technik (U.K) Limited under the Trade Name "CALLOTEK" and which is otherwise known as intercalated or oxygenated graphite. The intumescent material may comprise inert fillers or extenders, e.g. china clay, and/or reinforcing fillers, e.g. sugars, silicons, glasses and/or other granules, fibres or hollow spheres such as those sold under the Trade Name "VOLGLASS".

The intumescent material may comprise a flame retardant, e.g. aluminium trioxide or ammonium polyphosphate.

From another aspect the invention is a method of making an intumescent material comprising mixing granules or powder of a diene monomer and discrete particles of expandable graphite. Preferably the ingredients are mixed in a dry, or at least solid, state. The diene monomer may however be provided in slab form. The mixing should be such as to raise the temperature of the mixture to an extent to cause the diene monomer to become mechanically plasticised, to form a 'dough'-like compound e.g. in an open mill to which the particles of expandable graphite are added to form a homogenous mixture. The resultant mixture can then be stored or reprocessed, e.g. pelletised. Alternatively the mixture can be dissolved in a suitable solvent, e.g. xylene or toluene, to form a coating

composition. It is important that the flake structure of the graphite is not unduly damaged by the mixing, and we prefer to use open mill mixing using established rubber processing i.e. compounding, techniques and apparatus. It is also important that the mixing and/or pelletising does not raise the temperature of the graphite to such an extent to cause it to expand. Thus the temperature must be kept below around 190°C during this processing. In some circumstances it may not be necessary to take any steps during the mixing to induce cross-linking of the monomer, although in other applications, cross-linking of the binder may be advantageous, in which case a cross-linking agent can be added during mixing.

From yet another aspect the invention is a method of making an intumescent seal comprising the step of co-extruding a novel intumescent material as described above and one or more thermoplastic materials, e.g. polyvinylchloride.

A variety of proportions can be used to tailor the material to its particular application.

A number of additional ingredients can be added to enhance particular aspects of the material for processing, handling, assembly and when activated, although plasticity can be modified by using different molecular weights of the base monomer.

Whilst the polynorbadene is self-extinguishing, it is possible to modify the materials texture or performance by, for instance, adding a flame retardant, e.g. aluminium

trioxide or ammonium poly-phosphate. A preferred material comprises 50 parts expandable graphite, 50 parts polynorbadene and 10 parts aluminium trioxide, all by volume.

The material can be shaped by using established rubber and plastics processing techniques and machinery. The main reservation is the control of damage by mastication to the graphite, and confinement to temperatures less than that which induces expansion i.e. to a temperature of less than about 190°C.

BEST MODE FOR CARRYING OUT THE INVENTION

An example of use of the intumescent material would be in fire door seals, say thirty minutes; such seal would have a cross section of approximately 1.5_{mm} x 7.5_{mm} and would require a 50:50 ratio of ingredients, by volume. It must be borne in mind that the expansion characteristics of the graphite, usually in excess of 30 times its own volume, must also be considered.

The intumescent material may be for co-extrusion in a PVC extruder, together with PVC but other processes may be used, e.g:-

1. Production of mill sheet (10_{mm} x 1_{m} x 600_{mm} approx) for large pipe collars and further processing.
2. Calendaring for accurate sheet of various thicknesses, widths and lengths; typically 1.8_{mm} thick, 1_{m} wide and 21_{m} long.

   2.1 The frictioning of elastomers onto suitably treated woven cloths using calendars is of value

since the addition of cloth into a "sandwich" of the compound moderates scarification, not unlike a groyne on a beach, in a fire situation, the scarification being caused by convection currents. Ceramic and glass fibres are good examples although it is not necessary for the textile to resist the fire merely to moderate or delay the scarification of the exfoliated compound.

2.2 Frictioned or laminated sheet including cloth can be used to extend considerably the performance of many fire seals by producing a seal cross-section not unlike a swiss-roll, the compound and textile on the outside imbuing longevity on the successive inner layers.

3. Extrusions for non sheet-derived profiles using the minimum of damage to the material due to mastication. A large diameter, e.g. of 32_{mm}, slow screw extruder or a ram extruder would satisfy these requirements.

3.1 Extrusion may also be used in conjunction with other appropriate materials, e.g. foams or elastomers to produce co-extruded products.

3.2 The use of a pre-former (e.g. a ram-extruder manufactured by Barwell International of Swavesey, Cambridge, GB with a shearing facility at the die face) would enable both single and co-extruded products to be produced in very short lengths e.g. pellets as used in the PVC industry.

4. Sheets of the compound can be glued to a variety of other materials to complement the intumescence.

4.1 Foam, commonly zero-rated (A.S.T.M.S) and polyurethane can be laminated with the compound, with or without textile included to provide a fire seal for expansion joints in a variety of locations. A layer of very high density material e.g. lead can also be laminated with the foam and compound to offer both sound proofing and a fire seal (commonly used in maritime engine rooms).

4.2 The performance of glass or rock fibre decking (often called "batts") can be enhanced by one or both faces being coated with sheet(s) of compound with or without textile reinforcing.

4.3 Any number of shaped "gaskets" can be used for fire sealing; these can be cut from a sheet of the compound and the scrap/off cuts simply reprocessed. Not unlike other reprocessable materials e.g. PVC it is not wise to reprocess the same sheet too many times as the requisite mastications will degrade the fire performance.

4.4 Sheet or "pre-formed" compound can be shot moulded to three dimensional shapes, which the material will retain indefinitely without loading to provide or enhance seals for complex shapes, e.g. concealed door closers and other door furniture.

4.5 Cosmetic (e.g. PVC or veneered) sheeting or

enhancing (e.g. glass or aramid) sheeting can be "glued" to the compound for use a panels or cut into strips for use as fire seals for various applications.

This invention enables the production of a variety of products whose shape need no longer be dictated by the intumescent employed within it, e.g. air conditioning ducting fire barrier ventilators need no longer be rectangular, but can be co-extruded in the most aerodynamic shape desired.

This invention not only overcomes the common problem of hygroscopicity, but the material can be immersed in water and still be activated. This makes it particularly appropriate for use in hot and humid climates, damp and frosty (for exterior doors) climates and where the ingress of moisture might support undesirable organisms, e.g. in hospitals.

Also marine applications.

The compound remains flexible under a wide range of temperatures (-20C to 100C) and therefore lends itself to being formed in any application. Whilst there is no danger of a break or crack this would not jeopardise the integrity of the compound. Only the volume present and the method of retention is relevant.

The ability to co-extrude in a variety of shapes with a variety of materials not only enables the production of the industry standard FS30 (Fire and Smoke Seal 30 minutes) and FS60, but will also enable production of the

increasingly specified surface mounted seals for use with steel fire doors and retrofitting without recourse to routing i.e. rebating.

Given that glass and rock "wool" is becoming less frequently specified, for health and environmental reasons, and alternative method for upgrading partitioning becomes desirable. Also given the "pre-formed" co-extrusion of a "zero-rated" foam core with a layer of compound around the circumference and cut to short lengths; this could be blown into cavities (using similar technology to exterior wall and loft insulation) causing minimal disturbance, no health risk and significantly reduced costs.

Having co-extruded, for instance, an FS30 seal, it becomes a practical proposition to complete the manufacture of the product on the line i.e. adding the "cold smoke seal" the "double-sided self adhesive tape" and the inkjet printing of a logo; certification numbers, and ISO 9000 traceability data. Given the nature of the product and the life or death implications, automatic construction offers a higher level of confidence that there is actually intumescent compound throughout the length of the seal, however this can also be checked automatically on line using inductive proximity sensors tuned to the required amount of compound.

INDUSTRIAL APPLICABILITY

The present invention thus provides simple and effective intumescent materials.

CLAIMS

1. An intumescent material comprising expandable graphite and characterised by a binder incorporating a diene monomer.

2. An intumescent material according to claim 1, characterised in that the diene monomer is polynorbadene.

3. An intumescent material according to claim 1 or claim 2, characterised in that the expandable graphite is intercalated or oxygenated graphite.

4. An intumescent material according to any one of claims 1 to 3, characterised by inert fillers or extenders, e.g. china clay, and/or reinforcing fillers, e.g. sugars, silicons, glasses and/or other granules, fibres or hollow glass spheres.

5. An intumescent material according to any preceding claim, characterised by a flame retardant, e.g. aluminium trioxide or ammonium polyphosphate.

6. A method of making an intumescent material characterised by mixing a diene monomer and discrete particles of expandable graphite.

7. A method according to claim 6, characterised in that the ingredients are mixed in a dry, or at least solid, state.

8. A method according to claim 7, characterised in that the diene monomer is in granular or powder form.

9. A method according to any one of claims 6 to 8, characterised in that the mixing is such as to raise the temperature of the mixture to an extent to cause the diene

monomer to become mechanically plasticised, to form a 'dough'-like compound to which the particles of expandable graphite are added to form a homogenous mixture without raising the temperature of the graphite to such an extent to cause it to expand.

10. A method according to claim 9, characterised in that the homogenous mixture is pelletised.

11. A method according to claim 9, characterised in that the homogenous mixture is dissolved in a solvent, e.g. xylene or toluene, to form a coating composition.

12. A method according to any one of claims 6 to 11, characterised by open mill mixing of the ingredients.

13. A method according to any one of claims 6 to 12, characterised by maintaining the temperature of the expanded graphite below around 190°C during mixing.

14. A method according to any one of claims 6 to 13, characterised by the addition of a binder cross-linking agent during mixing.

15. A method of making an intumescent fire seal characterised by the step of co-extruding an intumescent material as claimed in any one of claims 1 to 5 and at least one thermoplastic material.

Patent Office

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Claims searched: 1 to 15 **Date of search:** 23 January 1998

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Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

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Other: ONLINE: CHABS, CLAIMS, JAPIO, RAPRA, WPI

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
X	GB 2212505 A	DIXON INTERNATIONAL see page 2, lines 13 to 28, page 4, lines 11 to 16, and Examples 4 and 5	1,3,4,6 at least
X	EP 0388481 A1	ZHEJIANG CI XI SEAL MATERIAL see the claims and column 2, line 42, to column 3, line 7	1,3,4,6,7, 9,13,
X	EP 0009109 A1	CHEMIE LINZ see the Examples	1,3,4,5,6, 7,13
X	US 4971726 A	LION CORPORATION see the claims, column 3, lines 10 to 20 and 63 to 68, and Example 4	1,3,4,5,6, 7,14
A	WPI Abstract Accession No. 94-132091/199416 & JP 060080768A (NIPPON ZEON) 22 March 1994 see abstract		

X Document indicating lack of novelty or inventive step
Y Document indicating lack of inventive step if combined with one or more other documents of same category.
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